

Compilation of Data from ORNL Characterization of German Reference Fuel Composite EUO 2358-2365

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This document was prepared to document the distribution of additional data summarized in a previous report entitled "Results from ORNL Characterization of German Reference Fuel from the EUO 2358-2365 Composite," ORNL/TM-2005/546 (also known as ORNL/CF-04/06), April 2004. This previous report was a compilation of the characterization data obtained on a sample of TRISO-coated 500 µm diameter UO₂ produced in Germany and obtained by the AGR program for use as a historical reference material. This sample came from the EUO 2358-2365 composite studied by General Atomics (GA) and referenced in GA document #910852 "Acceptance Test report for German Fuel Particles." The ORNL designation for the material characterized was AGR-06.

This update includes the individual particle data from measurements of kernel diameter, particle diameter, and coating thickness. This individual particle data was not included in the previous report, which only contained the summary data from the image analysis.

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1 Kernel Size and Shape

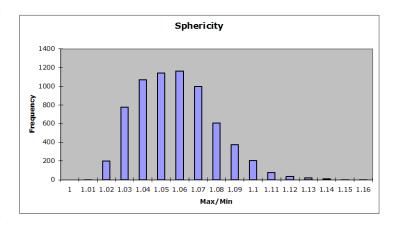
Shadow images for a random orientation of 6704 kernels (about 4.7 g) extracted from AGR-06 were obtained. Image analysis software was used to find the center of each kernel projection, identify 360 points around the perimeter, and measure the distance from center to each perimeter point (defined as the kernel radius). The uncertainty for this measurement was $\pm 1~\mu m$. This data was then compiled to report sphericity (maximum radius/minimum radius), mean diameter, standard deviation in diameter, maximum diameter, and minimum diameter for each kernel measured. Note that image analysis actually measured the radius of the kernel. The radius was multiplied by two in order to report the data in terms of diameter. This was done because these values are usually specified and reported in terms of diameter. The error introduced by making this conversion was small because the kernel cross sections were fairly symmetrical. The summary data from each kernel in the sample was then compiled to obtain the average, standard deviation, maximum, and minimum of the individual kernel quantities (sphericity, mean diameter, standard deviation in diameter, maximum diameter, and minimum diameter). Figure 1-1 contains the summary data and shows the distributions of the kernel sphericity and mean kernel diameter.

The measured kernels had an average mean diameter of 506 μm with a standard deviation in the distribution of 8 μm . The distribution was close to Gaussian. Based on variable sampling statistics, the average mean diameter of the German kernels is predicted to be 504 - 508 μm with 95% confidence. Less than 1% of the kernels measured had mean diameters outside the range 485 - 525 μm . The largest kernel measured had a mean diameter of 565 μm . The smallest kernel had a mean diameter of 472 μm .

Table 1-1 shows the output obtained from the image analysis software for the first 55 kernels analyzed. This table is an embedded Excel file. Double click the table to access the full data set for all 6704 kernels.

	Sphericity	Mean Diameter	St. Dev. In Diameter	Maximum Diameter	Minimum Diameter
Average	1.05	506	6	517	492
Standard Deviation	0.02	8	3	7	11
Maximum	1.16	565	17	571	555
Minimum	1.01	472	1	488	447

Sphericity	Frequency
1	0
1.01	2
1.02	203
1.03	779
1.04	1069
1.05	1143
1.06	1163
1.07	995
1.08	610
1.09	377
1.1	208
1.11	79
1.12	35
1.13	19
1.14	9
1.15	2
1.16	3
More	0



Mean Diameter	Frequency
470	0
475	3
480	3
485	11
490	88
495	414
500	1036
505	1584
510	1728
515	1108
520	513
525	162
530	34
535	8
540	3
545	0
550	0
555	0
560	0
565	1
More	0

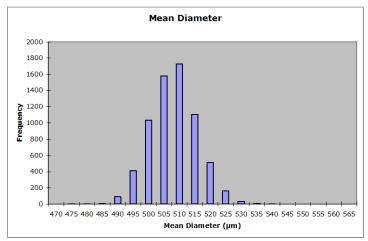


Figure 1-1. Size and shape summary for kernels extracted from German reference fuel. Reported diameters are actually two times measured radii. Diameters are in μm .

Table 1-1. Size and shape data for each individual kernel used to generate Figure 1-1 (double click to open this embedded Excel file)

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File	SubIm No.	Sphericity	Mean Diam.	Std. Dev. of Diam.	Max Diam.	Min Diam.	Fit Diam.
P04030408 XYF01 ZF0.tif	1	1.093619	504.89583	10.227372	530.72405	485.29176	504.9989
	2	1.060376	497.13847	6.485205	518.51756	488.99407	497.18058
P04030408 XYF02 ZF0.tif	1	1.02221	504.89951	1.53611	509.47023	498.40054	504.90184
	2	1.046266	496.1989	6.63869	507.12762	484.70249	496.24315
	3	1.027227	517.89237	2.615039	522.24236	508.39995	517.89895
	4	1.031305	517.67719	3.484266	525.65225	509.69616	517.68888
	5	1.050415	503.98466	6.394227	516.40514	491.62024	504.02506
	6	1.081187	505.01296	12.278178	527.25519	487.66312	505.16163
	7	1.048674	510.88619	7.751001	524.98354	500.61646	510.94479
	8	1.043298	501.6405	6.149001	511.42946	490.20458	501.67804
	9	1.036861	506.66612	4.65668	517.22347	498.83572	506.68744
	10	1.032035	509.79132	3.887635	515.05718	499.06938	509.80609
	11	1.022016	508.24948	1.478155	511.99115	500.96189	508.25162
	12		498.89973	3.75627	507.74223	489.023	498.91382
	13	1.034714	510.53906	2.794882	517.99903	500.62058	510.54668
	14	1.052633	495.89796	7.254715	507.90636	482.51032	495.95082
	15	1.046946	505.49254	5.254646	515.119	492.02082	505.51975
	16	1.033862	511.81889	3.53194	517.01066	500.07727	511.83103
P04030408 XYF03 ZF0.tif	10		492.38085	11.740108	517.01000	472.89786	492.52025
F04030408 X11 03 210.til	2		500.96231	9.406209	520.52909	485.87654	501.0502
	3	1.07132	505.88932	10.704726	522.82972	482.13432	506.00217
	4	1.050158	509.29726 506.55731	5.283195	520.02385	495.18651	509.32457
	5	1.059354		6.047594	518.67438	489.61369	506.59328
	6	1.046514	514.12691	6.771492	527.14584	503.7159	514.17136
	7	1.120532	501.46982	13.78052	522.10963	465.94817	501.65848
	8	1.075004	514.60376	6.986581	529.29827	492.36845	514.65101
	9	1.061977	505.99116	8.402347	521.64655	491.20311	506.0607
	10	1.06469	502.72404	6.608042	518.19982	486.71438	502.76732
	11	1.053165	496.92926	8.160994	508.63932	482.96247	496.99601
	12		499.89543	4.521664	509.45958	492.53175	499.9158
	13	1.093935	494.56426	14.410522	512.41143	468.41112	494.7734
	14	1.064147	502.3383	7.623888	519.31846	488.01364	502.39596
	15	1.077573	492.5318	10.697502	509.14025	472.48776	492.64754
	16	1.110552	508.46913	12.123493	528.3151	475.72296	508.61314
	17	1.066454	491.97653	9.723932	509.38095	477.63993	492.0723
	18	1.091191	500.51227	9.344691	517. 449 61	474.20609	500.59915
	19	1.051781	507.64529	6.294949	520.12931	494.52253	507.68418
	20	1.072056	503.9063	10.601654	520.90925	485.89765	504.01738
	21	1.052016	503.24069	6.485808	514.51979	489.07981	503.28234
P04030408 XYF04 ZF0.tif	1	1.075184	499.71504	8.060687	512.9847	477.11356	499.7798
	2	1.056468	504.17088	8.350919	516.15109	488.56286	504.23976
	3	1.055416	507.78356	7.337244	520.11996	492.81044	507.83639
	4	1.021799	517.4519	1.821596	520.61899	509.51212	517.45509
	5	1.059183	505.12285	7.770861	518.59746	489.62003	505.18241
	6	1.04554	512.42849	6.999074	523.08107	500.29735	512.47613
	7	1.045686	499.80354	4.889766	508.99926	486.76122	499.82738
	8	1.042022	513.20725	6.282087	522.98566	501.89503	513.24556
	9	1.036325	512.44228	5.468414	521. 0 6247	502.79844	512.47136
	10	1.033633	508.29061	4.653541	516.03957	499.24858	508.31184
	11		502.57658	4.137708	510.28549	491.80646	502.59355
	12		503.11373	10.328356	519.56113	487.03683	503.21938
	13	1.101052	496.12377	12.097914	517.33116	469.85183	496.27072
	14	1.029441	504.73531	3.000552	513.88714	499.1907	504.74419
	15		494.58989	13.072399	514.51102	467.93568	494.76191
	16		506.38138	7.709621	519.17069	493.65979	506.43985

2 Particle Size and Shape

Shadow images were obtained for a random orientation of 1118 coated particles (about 1.5 g) riffled from AGR-06. Image analysis as described above was used to measure the size and shape of the particles. Figure 2-1 contains the compiled data and shows the distributions of the particle sphericity and mean particle radius. In reporting the data for the particle size and shape, data was left in terms of the measured radius rather than multiplying by two to estimate the diameter, because the faceting of the coated particles was such that it was more appropriate to report the data in terms of radius.

The measured particles had an average mean radius of 461 μ m with a standard deviation in the distribution of 12 μ m. The distribution was close to Gaussian. Based on variable sampling statistics, the average mean radius of the German particles is predicted to be 460 - 462 μ m with 95% confidence. Less than 1% of the particles measured had mean radii outside the range 445 - 485 μ m. The largest particle measured had a mean radius of 501 μ m. The smallest particle had a mean radius of 425 μ m suggested that no particles measured are missing more than 80% of their 100 μ m thick buffer layer uniformly around the particle. However, the more aspherical particles exhibited local radii of as low as 391 μ m, which could indicate that more than 80% of the buffer is missing at that location.

Table 2-1 shows the output obtained from the image analysis software for the first 51 particles analyzed. This table is an embedded Excel file. Double click the table to access the full data set for all 1118 particles.

	Sphericity	Mean Radius	St. Dev. In Radius	Maximum Radius	Minimum Radius
Average	1.09	461	9	479	441
Standard Deviation	0.02	12	3	13	13
Maximum	1.18	501	18	524	486
Minimum	1.03	425	3	437	391
Sphericity	Frequency		Çı	phericity	
1	0		ار ح	bileficity	
1.01	0	250 -			
1.02 1.03	0 2	230			
1.03	2 11				
1.04	40	200 -		_	
1.06	85				
1.07	151				
1.08	168	150		1 	
1.09	196	<u>#</u>			
1.1	147	150 +			
1.11	119	_			
1.12	84				
1.13	44	50 +			
1.14	36				
1.15	23	0			, , , ,
1.16	7		0 0 0 0 0		N 6 6 1 6
1.17	2	, *tq.	to to to to		74 72 78 72 78
1.18	3			Max/Min	
More	0				
Mean Radius	Frequency				
425	0		Me	an Radius	
430	2				
435	6	200 T			
440	19	180			
445	54	160		∥п_	
450	113	140	П		
455	156				
460	180	100 - 20 120 -			
465	172	B 100 +			
470	158	₽ 80 +			
475	118	60 +			
480	59	40	ПШ		
485	46				
490	24	20 +			
495 500	9	0 +125	420, 425, 440, 445, 450, 455	460 465 470 475 100	105 400 405 500 505
500 EOE	1	425	430 435 440 445 450 455		185 490 495 500 505
505 More	1 0		·	Mean Radius (µm)	
More	<u> </u>				

Figure 2-1: Size and shape summary for 1118 coated particles. Radii are in μm .

Table 2-1. Size and shape data for each individual particle used to generate Figure 2-1 (double click to open this embedded Excel file)

File	SubIm No.	Sphericity	Mean Radius	Std. Dev. of Radius	Max Radius	Min Radius	Fit Radius
P04030402 XYF02 ZF0.tif	1		476.057	6.709139	488.5737	462.8724	476.1041
	2	1.066494	460.486	7.385698	475.6207	445.9667	460.545
P04030402 XYF03 ZF0.tif	1	1.127193	452.3778	14.235107	479.9725	425.812	452.6008
	2	1.124147	467.2137	10.3603945	485.0048	431.4424	467.3281
	3	1.084644	456.736	7.762316	471.8824	435.0572	456.8017
	4	1.110719	456.4353	14.1020185	478.5723	430.8671	456.6523
	5	1.107163	477.9464	10.3957315	498.1291	449.9146	478.059
	6	1.095265	458.0132	7.479883	474.4987	433.2274	458.074
P04030402 XYF04 ZF0.tif	1	1.127191	464.8409	12.8268425	485.6651	430.8632	465.0171
	2		444.6854	9.2817225	463.7294	428.0221	444.782
	3	1.078973	446.8466	7.654294	464.3847	430.3951	446.9118
	4		479.8916	9.133404	496.7388	458.8565	479.9781
P04030402 XYF08 ZF0.tif	1		457.7417	10.794947	476.5529	431.0399	457.8685
	2	1.120519	460.5803	11.4865765	482.6681	430.7542	460.723
	3	1.117856	471.3956	10.198023	497.5962	445.1345	471.5053
	4	1.066554	456.3504	7.135804	468.648	439.4039	456.4059
	5	1.070871	451.0937	6.977458	467.4373	436.5018	451.1475
	6	1.041726	454.0154	5.0848375	462.9535	444.4101	454.0437
P04030402 XYF09 ZF0.tif	1	1.121846	468.4806	11.938383	496.461	442.5395	468.632
	2	1.05567	447.3196	6.221019	457.3986	433.278	447.3627
	3	1.080651	457.0554	7.7436615	475.163	439.7007	457.120 7
	4	1.07057	490.9734	5.9387005	500.9455	467.9242	491.0091
	5	1.110747	454.6248	11.4712505	478.5043	430.7952	454.7689
P04030402 XYF10 ZF0.tif	1	1.042979	468.7098	5.6869815	480.2258	460.4368	468.7442
	2	1.086	466.547	8.4544665	488.2322	449.5692	466.6233
	3	1.130585	459.157	12.320141	477.5016	422.3493	459.3216
	4	1.092783	465.1548	8.265505	479.3044	438.6088	465.2279
	5	1.0896	453.2923	8.9470155	480.0785	440.6007	45 3.3 80 3
	6	1.078159	452.6193	10.477622	473.0916	438.7958	45 2.7 40 1
P04030402 XYF11 ZF0.tif	1	1.082104	461.4408	6.96872	477.0918	440.8929	461.4933
	2	1.097888	445.3866	8.361263	468.8123	427.0128	445.4647
	3	1.103018	466.6021	11.3096915	493.6273	447.5243	466.7388
	4	1.097511	481.4427	11.8618895	499.0271	454.6899	481.5884
	5	1.08899	455.3609	12.14 0 7135	468.4085	430.1312	455.5221
	6	1.105661	452.2231	8.6466905	464.2631	419.8964	452.3055
	7	1.093155	452.5437	9.912766	471.2372	431.0801	452.6519
P04030402 XYF12 ZF0.tif	1	1.059667	464.0816	5.9570305	476.1994	449.3857	464.1197
	2	1.128858	481.0241	15.7953615	507.2235	449.3243	481.2825
	3	1.067318	462.9661	8.7991985	478.3673	448.1957	463.0495
	4	1.091404	463.9421	9.9772415	486.9821	446.1977	464.049
P04030402 XYF15 ZF0.tif	1	1.069165	441.9537	7.476276	455.5877	426.1153	442.0167
	2		456.3555	5.8471425	469.0565	446.3299	456.3928
	3		461.6039	14.4181845	488.4095	428.1024	461.8283
	4		452.9003	7.634278	473.7551	438.5997	452.96 44
	5		467.2145	16.232442	495.8939	442.3228	467.4956
P04030402 XYF16 ZF0.tif	1		456.8207	7.378523	472.7 0 69	443.6604	456.88
	2		468.7252	10.655172		447.1104	468.8458
	3		459.8689	7.378624	474.9367	435.9842	459.9278
	4		447.0264	11.522672	465.7762	431.2725	447.17 44
	5		443.85	9.3034285	465.2901	426.7136	443.9471
	6	1.080277	466.6542	8.6037185	480.1968	444.5125	466.7333

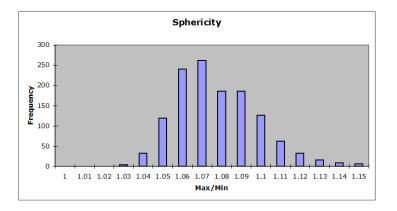
3 Particle Size and Shape After Removing OPyC

The same sample of particles described in section 2 was remeasured after removing the outer pyrocarbon (OPyC) layer by heating in air at 800°C for 4 hours. Figure 3-1 shows the data summary for 1283 particles measured. The measured particles had an average mean radius of 426 µm with a standard deviation in the distribution of 12 µm. The distribution was close to Gaussian. The difference in the average mean radius before and after OPyC removal was 35 µm. Direct measurement of the OPyC thickness as described in section 4.5 yielded an average mean thickness of 36 µm. The sample of particles before and after removal of the OPyC exhibited the same standard deviation in mean radius. This was expected for an OPyC layer with a standard deviation in thickness of a few microns. Note that the number of particles measured in Figure 2-1 was 1118. These numbers were different because the technique used did not measure every particle in the sample. Some particle projections were cutoff at the edges of the images and not analyzed.

Table 3-1 shows the output obtained from the image analysis software for the first 54 particles analyzed. This table is an embedded Excel file. Double click the table to access the full data set for all 1283 particles.

	Sphericity	Mean Radius	St. Dev. In Radius	Maximum Radius	Minimum Radius
Average	1.07	426	7	440	410
Standard Deviation	0.02	12	2	12	12
Maximum	1.15	462	15	495	450
Minimum	1.02	366	2	383	349

Sphericity	Frequency
1	0
1.01	0
1.02	0
1.03	4
1.04	33
1.05	119
1.06	241
1.07	262
1.08	186
1.09	186
1.1	127
1.11	62
1.12	32
1.13	16
1.14	9
1.15	6
More	0



Mean Radius	Frequency
365	0
3 70	1
375	0
380	0
385	0
390	0
395	0
400	9
405	33
410	60
415	124
420	182
425	218
430	212
435	171
440	119
445	86
450	39
455	23
460	5
465	1
More	0

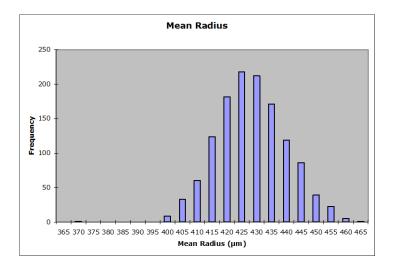


Figure 3-1: Size and shape summary for 1283 particles with the OPyC removed. Radii are in μm .

Table 3-1. Size and shape data for each individual particle used to generate Figure 3-1 (double click to open this embedded Excel file)

File	SubIm No.	Sphericity	Mean Radius	Std. Dev. of Radius	Max Radius	Min Radius	Fit Radius
P04040507 XYF02 ZF0.tif	1		425.02595	11.068952	442.70885	404.73596	425.1696
P04040507 XYF03 ZF0.tif	1	1.043951	433.95711	4.8434545	442.38761	423.76282	433.98406
	2		413.07052	9.4004785	430.1554	399.4988	413.17715
	3		441.23914	4.8866245	451.04065	429.30743	441.26612
P04040507 XYF04 ZF0.tif	1	1.079301	426.35498	6.741027	446.85077	414.01881	426.40806
	2	1.07359	412.80502	5.8924975	429.12725	399.71229	412.84689
	3	1.052352	432.50644	5.3704625	445.20322	423.05543	432.5396
P04040507 XYF08 ZF0.tif	1		425.13244	5.8978965	436.64621	413.9526	425.1732
	2	1.082097	424.0666	8.680693	441.51089	408.01419	424.15515
	3	1.085237	440.69744	8.6069845	454.15623	418.48571	440.7812
	4	1.095002	440.09683	10.554811	461.91212	421.83697	440.22296
P04040507 XYF09 ZF0.tif	1	1.104085	410.02988	10.22279	434.58183	393.61254	410.15682
	2	1.071588	414.62492	5.2070935	427.93748	399.34905	414.65747
	3	1.08393	417.32443	7.05619	433.49302	399.92726	417.38377
	4	1.059281	417.15927	5.2366355	429.06949	405.05744	417.19202
	5	1.085587	426.34008	10.7314005	447.14681	411.89415	426.47456
P04040507 XYF10 ZF0.tif	1	1.100649	426.84577	10.4236385	443.09063	402.57199	426.97263
	2	1.067305	422.90079	7.656638	435.34304	407.88981	422.9699
	3	1.040287	428.77268	3.962325	435.9456	419.06271	428.79093
	4	1.068719	420.21839	7.785516	432.0623	404.28072	420.29028
P04040507 XYF11 ZF0.tif	1	1.056149	425.84749	5.788881	438.5905	415.27321	425.8867
	2	1.061089	430.19364	7.1349015	441.77165	416.33803	430.25261
	3	1.053729	444.26144	6.469339	456.83157	433.53787	444.30839
	4	1.068081	445.78616	5.658782	453.8203	424.89307	445.82195
	5	1.050804	410.06351	5.3625575	421.60719	401.2236	410.09847
	6	1.064452	438.12734	5.3975665	446.57862	419.5385	438.16048
P04040507 XYF12 ZF0.tif	1	1.063276	430.52083	6.200215	445.96791	419.42815	430.56532
	2	1.133354	422.5239	11.772757	435.43797	384.20311	422.6873
	3	1.092553	403.77112	7.313803	416.41582	381.14013	403.83715
P04040507 XYF14 ZF0.tif	1		427.97737	7. 0573 22	445.61357	410.30781	428.03534
	2		434.97407	9.353054	451.47854	416.65815	435.07428
	3		428.65094	5.664145	437.93922	415.86841	428.68824
	4		433.10394	11.34752	449.26664	402.66276	433.25209
P04040507 XYF15 ZF0.tif	1	1.034748	436.03121	3.29503	443.94977	429.04131	436.04361
_	2		453.24903	10.018822	472.6677	428.63555	453.35934
P04040507 XYF16 ZF0.tif	1		438.36426	6.311705	448.16869	425.7518	438.40957
	2		420.8386	4.9854875	431.92905	409.78428	420.86805
	3		426.96115	5.5133065	436.55065	412.06506	426.99664
D0 40 40 F0 7 10 F4 7 7 F0 115	4		409.90569	3.523531	418.47974	403.60373	409.92079
P04040507 XYF17 ZF0.tif	1	1.05042	448.32384	5.610234	461.51979	439.36688	448.35884
	2		422.9925	7.206442	433.65327	405.47594	423.05367
	3		420.93218	8.68337	431.31959	402.13053	421.0214
	4		408.11096	7.654458	423.16033	390.59366	408.18251
D04040E07 VV/E10 7E0 ±£	5		414.6996	2.0713935	420.55621	410.85509	414.70475
P04040507 XYF18 ZF0.tif	1		431.07801	6.8131275	443.45106	418.13065	431.13165
	2		414.85657	8.3324325 6.026484	433.54801	399.99313	414.93999
	3		441.59156		453.43509	430.52779 391.39618	441.63254
P04040507 XYF19 ZF0.tif	4 1		404.24604 421.43413	6.1322985	418.01006 437.81548	391.39618 407.02946	404.29238 421.49013
FUTUTUJU / ATFIJ ZFU.UI	2		421.43413	6.8825495 9.616266	437.81348	397.25538	421.49013
	3		411.05808	5.8652135	427.51812	412.91109	411.77062
	4		445.78488	10.1905795	465.17932	424.18307	445.90096
	5		424.48449	6.36345	435.78204	410.98003	424.53204
	6		418.43423	6.6625165	437.43972	409.33676	418.48709
	v	1.000033	110.73723	3.0023103	137.73372	107.55070	110.70703

4 Coating Thickness

Coating thicknesses were measured on 186 particles by mounting particles in a clear epoxy and grinding and polishing the particles to close to, but not beyond, the midpoint. The polished cross sections were imaged with bright field reflected mode with a computer-automated optical microscope and the images were computer analyzed to extract the thickness info for each layer. The deviation of the measured layer thickness from the actual layer thickness due to the polished cross section not being exactly at a midplane was corrected by measuring the outer diameter of the particle and applying a geometric correction.

4.1 Kernel diameter

The cross section measurement provides a secondary measurement of the kernel diameter. This measurement introduces more uncertainty than the shadow image technique in section 1 because of some uncertainty in the kernel buffer interface created during grinding and greater effect from error introduced by correction to midplane. Figure 4-1 shows the data summary for the kernel radius. The average mean radius was 257 μ m. The mean kernel size measured by this technique was slightly higher than what was measured by shadow imaging (253 μ m). This error could also be related to a real gap between the kernel and buffer that may be included in the cross sectioned kernel radius.

Table 4-1 shows the output obtained from the image analysis software for the first 50 particles analyzed. This table is an embedded Excel file. Double click the table to access the full data set for all 186 particles.

1	Sphericity	Mean Radius	Stnd. Dev. In Radi	us Maximum Radius	Minimum Radius
Average	1.036	257	2.4	262	253
Standard Deviation	0.016	8	1.2	8	9
Maximum	1.101	279	7.4	285	272
Minimum	1.007	229	0.5	231	227
Sphericity	Frequency	•			
1	0			Histogram	
1.01	1				
1. 0 2	27				
1.03	51				
1.04	44	40 +			
1.05	32	30 +			
1.06	15	W 20			
1.07	12				
1.08	3				
1.09	1				
1.1	0		1.01 1.02 1.03	1.04 1.05 1.06 1.07 1.03	8 1.09 1.1 1.11
1.11	1			Sphericity	
More	0	_			
Mean Radius	Frequency	-			
230	1	-		Histogram	
235	0	,			
240	4	. 60 T			
245	7	50 +			
250	25	40 +			
255	29	70 - 40 - 20 - 20 - 20 - 20 - 20 - 20 - 2			
260	54	20 +			
265	38	10 +			
270	16	0		, , , , , ,	
275	10	230	235 240 245	250 255 260 265	270 275 280
280	3			Mean Radius	
More	0				

Figure 4-1: Data summary for kernel radius from cross section measurement. Radii are in μm.

Table 4-1. Kernel radius data for each individual particle used to generate Figure 4-1 (double click to open this embedded Excel file)

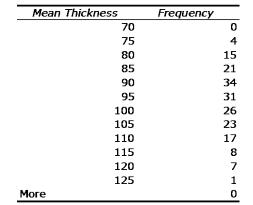
File	Krnl Sphricity	Krnl Mean	Kml Std	Kml Min	Kml Max
P04021203n01m02.tif	1.04908248	258.622577	3.885165	252.229893	264.609961
P04021203n01m03.tif	1.03805792	272.193351	3.179823	267.206935	277.376276
P04021203n01m04.tif	1.10067721	242.417913	7.386056	232.760723	256.194424
P04021203n01m05.tif	1.02572696	261.007578	1.916326	257.790971	264.42315
P04021203n01m06.tif	1.04029595	246.134644	2.86659	241.393864	251.12106
P04021203n01m07.tif	1.04248431	256.284875	2.563064	249.774285	260.385772
P04021203n01m08.tif	1.02569184	260.877358	1.654998	258.143426	264.775606
P04021203n01m09.tif	1.01843285	267.452266	1.061275	263.854923	268.718521
P04021203n01m10.tif	1.04734005	248.192625	3.79171	242.834069	254.329847
P04021203n02m01.tif	1.06897151	258.013034	5.398413	250.011432	267.255099
P04021203n02m02.tif	1.01895481	259.267757	1.309241	256.589094	261.452692
P04021203n02m03.tif	1.02834015	252.772245	2.080318	249.62196	256.696285
P04021203n02m04.tif	1.05465616	248.599783	4.011549	242.687318	255.951676
P04021203n02m05.tif	1.02101395	255.404919	1.33123	252.48676	257.792504
P04021203n02m06.tif	1.02806178	271.541339	2.023967	267.854339	275.370809
P04021203n02m07.tif	1.03559582	252.767053	2.50216	248.425432	257.268338
P04021203n02m08.tif	1.03886926	255.75403	2.6228	250.254234	259.98143
P04021203n02m09.tif	1.03077167	248.520335	1.903903	244.265915	251.782385
P04021203n02m10.tif	1.06143055	260.285619	4.022836	251.911879	267.386964
P04021203n03m01.tif	1.04919874	257.72071	4.228075	251.633843	264.013911
P04021203n03m02.tif	1.06262571	254.715366	4.734097	247.104326	262.579411
P04021203n03m03.tif	1.03141936	256.942006	2.459422	253.302905	261.26152
P04021203n03m04.tif	1.03850209	255.956851	2.767598	252.640761	262.367958
P04021203n03m06.tif	1.02829932	269.796624	1.432776	265.60607	273.12254
P04021203n03m07.tif	1.03265193	261.695011	2.238614	257.282155	265.682915
P04021203n03m08.tif	1.02918211	260.504052	1.935528	257.571154	265.087624
P04021203n03m09.tif	1.02346469	266.553173	1.74693	263.802047	269.992081
P04021203n03m10.tif	1.02459198	253.916541	1.837586	251.709499	257.899533
P04021203n04m01.tif	1.04953114	256.117996	3.455035	249.945156	262.325224
P04021203n04m02.tif	1.03421631	249.364669	2.472245	245.519233	253.919994
P04021203n04m03.tif	1.02495432	269.472068	1.907471	265.772786	272.404965
P04021203n04m04.tif	1.05365183	253.385966	3.597748	247.230321	260.494679
P04021203n04m05.tif	1.06905509	244.051268	3.931466	236.903253	253.262628
P04021203n04m06.tif	1.03492591	271.456558	2.570753	265.84991	275.134961
P04021203n04m07.tif	1.0299624	268.595918	1.843487	265.620035	273.57865
P04021203n04m08.tif	1.03996913	259.225929	3.247632	254.429881	264.599223
P04021203n04m09.tif	1.04251971	254.848698	2.669038	249.56629	260.177777
P04021203n04m10.tif	1.01560266	257.214212	1.04451	255.040331	259.019638
P04021203n05m01.tif	1.04518307	259.637798	3.262349	254.426625	265.922402
P04021203n05m02.tif	1.0189546	259.329632	1.073885	256.592016	261.455614
P04021203n05m03.tif	1.03564962	252.298755	2.393432	248.050476	256.893382
P04021203n05m04.tif	1.02715561	263.897907	1.722513	260.510583	267.584907
P04021203n05m05.tif	1.04220672	245.739802	2.749556	240.941298	251.110639
P04021203n05m06.tif	1.03234638	264.228554	1.809449	259.712531	268.113291
P04021203n05m07.tif	1.02243084	259.992892	1.028777	256.249396	261.997284
P04021203n05m08.tif	1.03614309	248.10763	2.37564	244.663809	253.506715
P04021203n05m09.tif	1.04780657	255.081388	3.620828	249.713007	261.65093
P04021203n05m10.tif	1.0246864	254.053004	1.505065	250.74674	256.936774
P04021203n06m01.tif	1.05682428	270.47486	4.283212	264.551341	279.584281
P04021203n06m02.tif	1.02602549	259.084715	1.52296	254.833979	261.466158

4.2 Buffer thickness

Figure 4-2 shows the data summary for the measurements made on the buffer. The average mean buffer thickness was 94 μm with a standard deviation in the distribution of 11 μm . The thickness distribution was close to Gaussian. Based on variable sampling statistics, the average mean buffer thickness of the German kernels is predicted to be 92-96 μm with 95% confidence. The thickest point measured in a buffer layer was 131 μm . The thinnest point measured in a buffer layer was 63 μm . Figure 4-3 shows an example of a buffer with a high standard deviation in thickness. This resulted in a faceted particle. The non-uniform buffer layer also resulted in more thickness deviation in the outer layers. Where the buffer layer was thinner, the other layers also tended to be thinner. The strong dependence on particle shape and coating uniformity on the buffer uniformity suggest that some optimization of the buffer coating step would be valuable. It would be interesting to study the correlation between irregularity in the buffer thickness and coating bed fluidization conditions.

Table 4-2 shows the output obtained from the image analysis software for the first 50 particles analyzed. This table is an embedded Excel file. Double click the table to access the full data set for all 186 particles.

	Mean Thickness	Stnd. Dev. In Thickness	Maximum Thickness	Minimum Thickness
Average	94	5.0	105	83
Standard Deviation	11	1.3	12	10
Maximum	121	9.1	131	108
Minimum	72	2.5	83	63



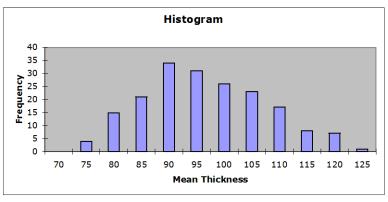


Figure 4-2: Data summary for buffer thickness. Thicknesses are in μm.

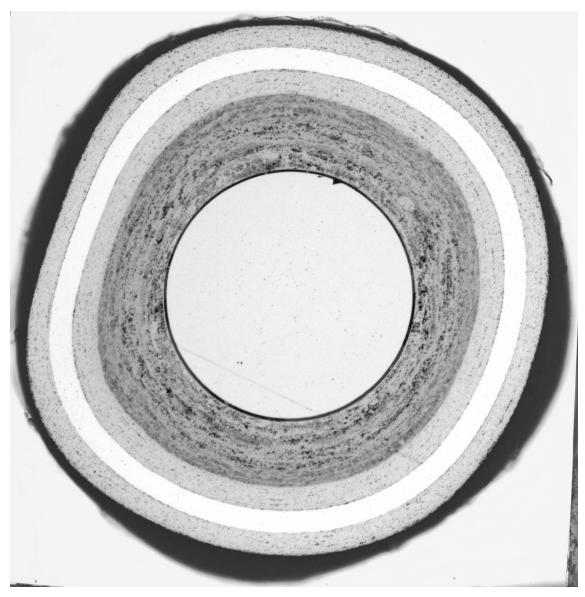


Figure 4-3: Particle with non-uniform buffer layer.

Table 4-2. Buffer thickness data for each individual particle used to generate Figure 4-2 (double click to open this embedded Excel file)

75.15	T	75.15	7
Bfr Mean	Bfr Std	Bfr Min	Bfr Max
107.566543	5.971299	93.062949	119.591667
87.786739	4.559726	76.925931	97.70676
113.252627	5.569895	101.550515	122.773489
87.80176	6.396931	76.697773	103.668635
89.570539	3.2028	83.850898	98.441693
95.044506	6.320131	81.290103	108.703111
101.92619	4.626278	91.625433	110.63768
96.213175	4.3165	87.289209	105.417166
94.822879	5.094574	84.976549	106.199523
84.482651	3.835016	76.273487	96.61217
99.848834	5.786573	88.32358	113.525861
92.267024	4.814646	81.806603	102.145287
101.399582	6.512489	87.216544	112.418825
105.967677	4.558546	93.598663	114.821637
79.679502	3.418662	73.969686	87.67619
104.678122	7.663498	84.185916	116.904667
115.734248	6.240527	104.759219	129.961501
101.294848	4.95116	88.518077	109.741051
88.267552	6.554568	73.062667	103.570692
90.821562	6.45159	76.696249	100.129949
117.45796	6.074128	100.641701	128.496854
89.113985	4.06447	81.26959	95.860385
105.423376	5.647759	93.573882	114.796856
93.525203	4.45592	85.485528	104.05563
76.147828	4.282684	67.371244	89.478508
93.187972	4.379701	83.506218	103.402756
96.295177	8.869631	80.61867	112.453131
91.177204	4.609179	82.269204	99.955016
86.808537	3.834239	77.506292	96.518539
88.904042	3.725334	82.036052	100.606154
86.044586	4.567917	76.793924	96.690462
98.506336	8.550626	84.889489	117.166095
119.002587	6.596308	105.6498	131.294226
91.852855	8.062751	77.006599	107.514624
80.155617	6.687548	69.24691	95.775627
84.623033	7.995184	66.402962	99.121714
108.573028	5.588782	92.380685	122.88871
93.20837	4.236848	85.445036	102.688702
78.862682	2.455847	72.273489	85.095702
95.77301	3.738525	86.311101	103.112622
96.862368	3.999282	87.431163	105.55912
92.274768	4.130851	84.610917	102.296728
75.820527	3.901708	68.961134	86.2048
107.660989	6.763216	97.963269	124.934131
79.778584	6.033072	66.967424	95.264722
110.67669	4.871109	99.101081	122.534781
78.868565	3.084077	71.078211	85.22686
96.444446	5.706659	84.494242	112.79154
74.160752	6.097321	64.259154	93.882889
78.392908	4.594181	68.014775	90.564185

4.3 IPyC thickness

Figure 4-4 shows the data summary for the measurements made on the inner pyrocarbon (IPyC). The average mean IPyC thickness was 39 μm with a standard deviation in the distribution of 3 μm . The thickness distribution was close to Gaussian. Based on variable sampling statistics, the average mean IPyC thickness of the German kernels is predicted to be 38-40 μm with 95% confidence. The thickest point measured in an IPyC layer was 56 μm . The thinnest point measured in an IPyC layer was 21 μm .

Table 4-3 shows the output obtained from the image analysis software for the first 50 particles analyzed. This table is an embedded Excel file. Double click the table to access the full data set for all 186 particles.

	Mean Thickness	Stnd. Dev. In Thickness	Maximum Thickness	Minimum Thickness
Average	39	2.5	46	33
Standard Deviation	3	0.5	3	3
Maximum	46	4.7	56	40
Minimum	33	1.4	37	21

Mana Thirtenan	<u></u>	
Mean Thickness	Frequency	11:-4
32	0	Histogram
33	2	
34	2	35 —
35	6	20
36	6	30 †
37	16	25 -
38	23	급
39	21	\$ 20 +
40	30	20 + 15 +
41	28	
42	25	10 +
43	14	
44	8	5 + N N N N N N N N N N
45	4	
46	1	32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47
47	1	Mean Thickness
More	0	

Figure 4-4: Data summary for IPyC thickness. Thicknesses are in μm.

Table 4-3. IPyC thickness data for each individual particle used to generate Figure 4-4 (double click to open this embedded Excel file)

IPyC Mean	IPyC Std	IPyC Min	IPyC Max
39.292943	2.82808	30.486882	46.404113
36.528772	2.162386	32.507707	41.792758
39.602955	2.367757	32.327209	45.149422
39.703507	3.298905	32.274238	49.075759
39.618814	2.279911	34.341318	45.837096
37.288326	2.601184	31.560088	43.055866
46.10931	2.9175 44	40.123154	52.503222
38.458947	1.964122	34.461217	44.188413
43.78064	2.294513	37.500949	50.323162
34.215271	2.052519	29.838033	40.44952
39.976318	2.430855	33.223777	47.372426
38.957025	2.555196	32.757165	45.137233
37.985056	2.384834	32.199093	43.694871
39.414073	2.886132	33.542138	45.480061
39.47255	2.220729	32.28032	46.871114
33.356848	2.361404	27.640892	39.578814
39.659268	2.630607	33.834004	45.771927
41.372877	2.651365	35.966423	47.4622
41.918531	2.856974	36.254158	48.634227
32.82145	2.097719	27.491143	38.10263
35.023907	2.354384	29.352166	39.963653
39.560508	2.023855	35.591026	46.644658
37.848281	2.842021	30.039503	43.303862
38.529236	2.049528	33.612826	43.782167
36.124329	2.361139	31.226342	44.932846
39.40954	2.391497	34.446459	44.173656
41.732628	4.072684	32.902004	51.914251
40.933124	2.539009	35.435784	48.257998
38.272559	2.260516	32.337987	43.391619
42.381375	2.445521	37.146866	48.200498
37.862637	3.112718	30.83007	45.420865
35.73376	2.290253	27.18439	41.775185
39.148237	3.615271	31.072945	49.200902
34.842418	3.151852	27.216639	42.249579
42.964458	2.632967	36.290521	48.670589
34.849382	2.288382	28.481262	40.86133
39.844372	3.181893	32.283688	51.73808
41.889661	1.87475	37.948427	46.791333
41.14042	2.980924	35.322525	48.586884
38.601735	1.758639	34.902452	44.629649
43.690325	1.734052	38.745666	48.472863
41.89376	2.178594	37.472307	49.41023
37.640731	1.87828	33.732658	42.575564
34.774509	2.59168	30.317439	40.48678
41.998047	2.257364	36.435613	48.373536
39.099077	2.94046	31.512601	45.66125
40.538383 43.695262	1.866551 2.878949	35.808657 37.672261	45.093708 50.936619
36.035259	2.878949	31.310446	44.132659
42.277801	2.718718	36.861521	50.568025
72.2//001	2.2013/8	20.001221	30.300023

4.4 SiC thickness

Figure 4-5 shows the data summary for the measurements made on the SiC. The average mean SiC thickness was 33.9 μm with a standard deviation in the distribution of 1.4 μm . The thickness distribution was close to Gaussian. Based on variable sampling statistics, the average mean SiC thickness of the German kernels is predicted to be 33-35 μm with 95% confidence. The thickest point measured in a SiC layer was 43 μm . The thinnest point measured in a SiC layer was 24 μm . The maximum standard deviation in thickness around a SiC layer was only 2.5 μm . This indicated that the SiC layers were very uniform in thickness on each particle. The largest local deviations in SiC thickness that were observed were associated with large deviations in the buffer thickness.

Table 4-4 shows the output obtained from the image analysis software for the first 50 particles analyzed. This table is an embedded Excel file. Double click the table to access the full data set for all 186 particles.

	Mean Thickness	Stnd. Dev. In Thickness	Maximum Thickness	Minimum Thickness
Average	33.9	1.3	37.1	31.0
Standard Deviation	1.4	0.3	1.7	1.7
Maximum	36.6	2.5	42.9	34.8
Minimum	28.9	0.7	31.4	24.2

Mean Thickness		Frequency	
	27		0
:	28		0
	29		1
	30		2
•	31		3
•	32		13
•	33		30
:	34		38
	35		57
	36		34
•	37		8
More			0

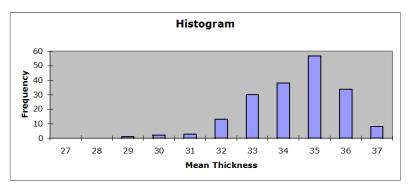


Figure 4-5: Data summary for SiC thickness. Thicknesses are in μm.

Table 4-4. SiC thickness data for each individual particle used to generate Figure 4-5 (double click to open this embedded Excel file)

SiC Mean	SiC Std	SiC Min	SiC Max
34.278922	1.374615	30.074857	37.149182
32.614657	1.21055	30.573419	36.763453
30.89053	1.233241	27.801654	34.433833
31.404841	1.957329	27.531157	37.258353
36.141873	1.273359	33.208975	38.956864
34.910676	1.211696	32.278683	38.026572
32.292685	1.946175	28.423914	38.15111
32.94456	1.114956	30.123428	35.871316
33.591952	1.223278	30.941537	36.689425
32.667473	1.192473	30.273748	36.021637
33.806678	0.917316	31.184511	36.490254
33.84454	1.006381	31.25799	36.121588
33.44661	1.461003	30.803564	37.435743
34.732413	1.193843	31.378249	38.010429
33.530932	1.184194	31.385299	36.248897
33.220164	1.505193	29.698968	37.215438
31.305191	1.258557	29.105518	35.737698
34.252746	0.917712	31.974469	36.395922
33.297525	1.254836	30.408842	36.156731
35.011872	1.272156	32.171088	38.361122
34.066819	1.06784	31.63502	36.940763
34.352251	1.044072	32.175913	36.597366
35.097609	1.46267	31.402011	38.476336
32.848993	0.916674	30.763541	35.184994
35.727184	1.519779	33.12344	39.755619
34.891669	1.280502	32.142999	37.890888
33.937679	1.631541	30.782481	37.856806
29.753145	1.15268	27.193615	32.499359
33.822834	1.353425	29.588065	36.662389
33.091282	1.335001	30.496134	36.686168
32.531426	1.607669	29.044619	35.234653
32.392325	1.345767	29.35012	35.540154
32.589904	2.010416	27.032383	36.759579
33.766709	1.838084	29.825475	39.552672
31.968808	1.44817	27.627186	35.143656
32.004759	1.110334	29.274512	34.580255
34.201781	1.264643	31.110 44 8	37.300482
34.010421	0.980599	32.453087	36.87454
35.065935	1.318954	31.501752	38.133932
34.981564	0.993154	32.843301	36.822608
35.634316	1.403602	32.326823	38.516857
33.24715	0.975076	30.76868	35.632279
33.344307	1.204316	30.364739	36.112628
34.068198	1.709521	30.726317	37.358496
35.148705	1.767657	32.273532	39.790002
32.34659	1.369813	27.751963	34.826288
34.633875	1.043126	32.247519	37.111117
36.331551	1.674115	32.842287	40.800903
35.818566	1.456792	33.085862	40.160187
35.369058	1.285595	32.694079	38.441967

4.5 OPyC thickness

Figure 4-6 shows the data summary for the measurements made on the OPyC. The average mean OPyC thickness was 36 μm with a standard deviation in the distribution of 2 μm . The thickness distribution was close to Gaussian. Based on variable sampling statistics, the average mean OPyC thickness of the German kernels is predicted to be 35-37 μm with 95% confidence. The thickest point measured in an OPyC layer was 51 μm . The thinnest point measured in an OPyC layer was 26 μm . Figure 4-7 and Figure 4-8 show that the thickest and thinnest regions observed in the OPyC layers were associated with faceted regions in the particle.

Table 4-5 shows the output obtained from the image analysis software for the first 50 particles analyzed. This table is an embedded Excel file. Double click the table to access the full data set for all 186 particles.

	Mean Thickness	Stnd. Dev. In Thickness	Maximum Thickness	Minimum Thickness
Average	36	2.6	42	30
Standard Deviation	2	0.5	2	2
Maximum	45	4.3	51	37
Minimum	32	1.4	36	26

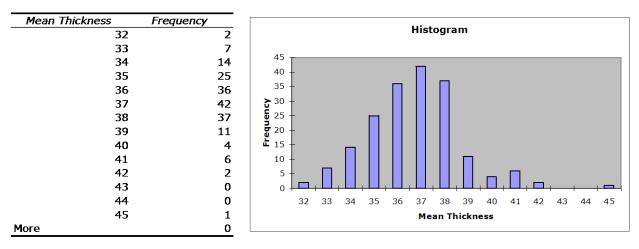


Figure 4-6: Data summary for OPyC thickness. Thicknesses are in µm.

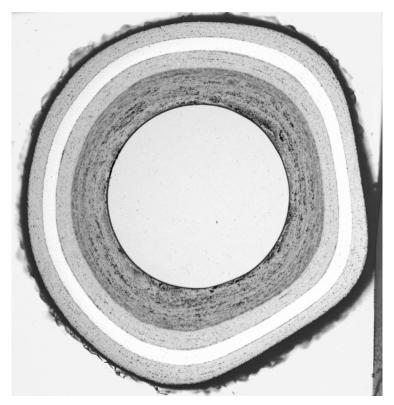


Figure 4-7: Particle showing thickest local OPyC region.

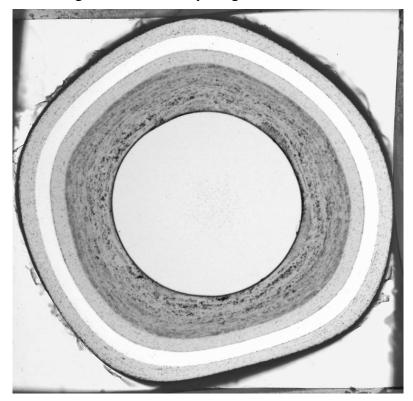


Figure 4-8: Particle showing thinnest local OPyC region.

Table 4-5. OPyC thickness data for each individual particle used to generate Figure 4-6 (double click to open this embedded Excel file)

OPyC Mean	OPyC Std	OPyC Min	OPyC Max
36.990771	2.866332	29.879601	44.470395
32.097883	2.277454	27.414827	38.026314
37.833067	2.676304	31.345813	44.168026
37.967975	4.303885	29.281049	49.177587
37.73655	2.844295	31.222276	43.602345
35.355789	2.24156	30.882753	41.49424
37.158814	3.285772	31.124759	44.389118
34.185987	2.120598	29.069384	38.79658
34.608883	2.024129	30.859246	39.702151
33.765371	2.724434	27.893436	42.484231
36.13182	1.51121	31.055746	39.456507
36.099909	2.49481	30.563267	40.732609
33.30066	2.295937	28.900087	39.069428
37.689369	2.206477	32.908059	43.077401
34.180859	2.442395	29.58132	39.308516
34.249124	3.253808	26.770728	40.477232
37.351049	2.633863	33.03522	44.973142
37.425131	2.417964	30.808918	41.86255
36.221537	2.296287	30.169059	42.106982
35.150544	2.471785	30.27712	40.888607
33.361721	1.844885	29.432769	37.391384
39.668299	2.503453	33.173676	46.438034
34.830565	3.288019	27.856951	41.12131
34.938073	2.329104	29.330197	40.383829
38.655709	2.268225	34.350934	43.635985
34.289318	2.255488	30.057005	39.784201
36.265552	4.031014	28.292199	44.209429
40.124591	2.546245	34.483554	46.421476
37.570273	3.305923	27.123363	43.040593
36.51606	3.013536	30.145483	43.409842
35.634925	3.089928	29.313475	42.577834
34.944203	3.084883	29.172979	42.879483
37.599715	3.341497	30.212204	44.360853
34.665102	3.597095	25.88729	42.688811
35.975582	2.888068	29.654133	41.592056
37.951547	3.166002	29.290412	44.323352
36.348523	2.539038	30.171998	43.436357
37.407913	2.221658	30.374118	43.196332
36.949491	2.585952	30.935087	41.988719
36.353877	2.166806	31.717492	42.328979
41.318169 37.453763	2.243393	32.938288	46.202647
	1.870566 3.053158	32.319965	41.16287
36.396971	2.396913	29.678819 26.962896	42.058888
32.312854 39.163235		33.859948	38.016528 44.471435
39.163235	2.43205 3.007212	29.830784	44.471435
36.401555	2.290478	29.83078 4 31.632776	41.802117
35.408443	2.903344	27.546854	42.579794
35.277424	2.622129	28.820875	42.969524
39.098743	2.658128	32.497268	44.877336
33.0307 T3	2.030120	32.137200	, , 550

4.6 Total particle radius

The mean kernel radius and mean layer thickness data were summed for each particle as a comparison check against the data obtained from the whole particle measurements made in section 2. The average mean radius obtained by summing the data from the cross section measurements was 461 μ m with a standard deviation in the distribution of 10 μ m (Figure 4-9). This agreed well with the data obtained by shadow imaging the whole particles summarized in Figure 2-1 (461 μ m with a standard deviation in the distribution of 12 μ m).

	Mean Radius
Average	461
Standard Deviation	10
Maximum	486
Minimum	429

Mean Radius	Frequency
430	0 1
43.	5 1
440	0 0
44.	5 5
450	0 19
45:	5 29
460	39
46.	5 29
470	30
47:	5 21
480	0 11
48	5 1
490	0 1
More	0

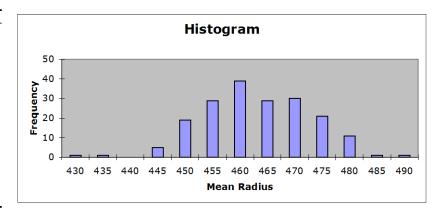


Figure 4-9: Data summary for total particle radius calculated from sum of kernel radius and layer thicknesses. Radii are in μm.